Environmental Protection Agency

Preparation of Gravimetric Binary Gas Mixtures

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NVFEL Reference Number

101B

Implementation Approval

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(1) 03-01-96 The purpose of this change is to revise the procedure as described in EPCN #172.

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1. Purpose

This procedure describes and illustrates the equipment required, the blending process, and the calculations used by National Vehicle and Fuel Emissions Laboratory (NVFEL) to generate binary gravimetric gas mixtures used as primary standards. The gravimetric method is considered to be the most accurate and reproducible technique for blending primary analytical gas standards.

Once gravimetric gas standards are blended according to this procedure, they must be successfully correlated with other gravimetric standards and National Institute of Standards and Technology (NIST) Standard Reference Material according to TP 403, Standard Gas Correlation. This must be completed prior to using the gravimetric standard to name secondary standards.

2. Test Article Description

Binary mixtures are prepared from pure components or by blending diluted mixtures of propane (C_3H_8), carbon monoxide (CO), carbon dioxide (CO_2), or methane (CH_4).

Ultra-high-purity zero-grade nitrogen or hydrocarbon free synthetic air is used for the dilution of these gas mixtures.

3. References

3.1 "Matheson Gas Data Book," 1971

3.2 "The Present State of the Art in the Preparation of Gaseous Standards," Scientific Gas Products, Inc., 1966

3.3 "Handbook of Compressed Gases," Compressed Gas Assoc., Inc., Reinhold Publishing Corp., New York, NY, 1966

3.4 Environmental Protection Agency (EPA) current safety policies

3.5 "Gas Laboratory Inventory Book"

4. Required Equipment

4.1 Form 101-01, "Weight Cross-check Worksheet" (Attachment B)

4.2 "Gravimetric Gas Blend Program"

4.3 Computer, Data Acquisition System, and Printing System

Equipment Used: Macintosh IIci with color monitor
Hewlett-Packard LaserJet printer
Gravimetric Gas Blending Program with Form 101-02

4.4 High Pressure Gas Cylinders

Equipment used: Carbon steel and alrock-coated aluminum (internal water volume of approximately 228.5 cubic inches)

4.5 Manifold Valves

Equipment used: CPI Valve - 4F-V6 LN-SS (three required)

Bellows Valve - Nupro SS4D 454 (four required)

4.6 Balance

Equipment used: Voland Model 1115 CDN
10 kilogram capacity
1 milligram readability
Reading device should be a center scale micro-amp meter

4.7 Working Weights (2 sets required)

Equipment used: 1 gram to 1 kilogram
Class M for standard set
Current calibration traceable to NIST Class S-1 or better

4.8 Blending manifold with a 0-2000 pounds per square inch actual (psia) pressure gauge and a 0-30 psia vacuum gauge
See Attachment C

4.9 Digital Pressure Controller (DPC)

Equipment used: Heise, Model No. 710A (two required)
0-1000 psia
0-2000 psia

4.10 Teflon Tape

4.11 Vacuum Pump, 150 liter/minute maximum capacity

4.12 Pure Gases:

Hydrocarbon-free grade synthetic air

Zero-grade Nitrogen, ultra-high purity - 99.999% purity

Propane, Research grade, 99.9% minimum purity

Carbon Dioxide, Coleman grade, 99.9% minimum purity

Carbon Monoxide, Total Hydrocarbon 1 ppm, 99.9% purity

Methane, ultra-high purity grade, 99.97% purity

4.13 Solenoids - two required

Equipment used: Peter Paul Electronics Inc. - Model 72Z0024GM

4.14 Smooth-gripping non-magnetic tweezers (for small weights)

4.15 Aluminum slot tool (for small weights)

4.16 Gas Laboratory Inventory Book

5. Precautions

5.1 Gas blending should be attempted by qualified personnel familiar with the chemistry of gases and the operation of the blending equipment . Equipment damage, serious injury, or loss of life could result from deviations from prescribed practices.

5.2 Personnel should be familiar with the safe handling of compressed gases.

5.3 Gas leaks must be avoided because of the toxicity of these compounds.

5.4 Avoid sudden pressure surges when blending or transferring gases. Always “bleed” gas slowly from one cylinder to another in order to minimize temperature changes.

5.5 Special precautions should be taken when blending combustible gases such as propane with air. If at all possible, use only parent concentrations of propane in nitrogen that are below the flammable limit in air.

5.6 Ensure that only the valve for the diluent gas being used is open.

5.7 All traces of combustible material such as oil, grease, and solvents must be removed from the gauges, fittings, valves, and tubing contained in the blending manifold.

5.8 All manifold parts must be specified "cleaned for oxygen service" when ordered.

5.9 All valves, unless otherwise directed shall be either fully opened or fully closed. The needle valves, V1 and V6, shall be operated only as the procedure describes.

5.10 The pressure of the diluent cylinder must always higher than the pressure of the cylinder being filled so as not to cause gas to back flow from the cylinder being filled to the diluent cylinder.

5.11 Never drop the gas cylinder or weights onto the balance pans or release the pans prior to the loading process.

5.12 Weights should only be handled with smooth-gripping, non-magnetic tweezers.

5.13 The weights shall be kept in their box or within the enclosed balance housing when not in use and shall only be handled with the transfer tools provided.

6. Visual Inspections

Visual inspections of the "Gas Blender" and "Voland Balance" are conducted before starting the test. Instructions for specific inspections are covered in Section 7, "Test Article Preparation."

7. Test Article Preparation

The following is a procedure to verify that the Voland Balance and weights used in the gravimetric blending are within NVFEL standards. This procedure is to be performed once every three months or just prior to doing gravimetric blending.

7.1 Remove all weights from the balance pans and case. Remove any dust and grease smears from the weights with a lint free cloth. Do not handle them with bare fingers.

7.2 Rotate the vernier mechanism knob (see Attachment A) to zero and slowly release the balance pans by rotating the “Pan Arrest” and “Beam Arrest” handles counterclockwise. Check the action of the pan arrest pads and, if necessary, adjust them to achieve smooth operation.

7.3 Observe the micro-amp meter on the right-hand side of the balance and allow the pans to stabilize. Stabilization occurs when there is no discernible needle movement.

Note: If the meter is not at center scale, the circuitry may be turned off. Press the red button above the vernier mechanism knob to turn it on. The indicating needle on the meter should be at center scale.

If the meter does not read center scale, adjust the “Zero” knob, located just below the meter, to bring the needle to the center scale position.

Rotate the “Pan Arrest” handle and “Beam Arrest” handle clockwise to lock and counterclockwise to unlock the pans several times to ensure that the meter stabilizes in the center scale position.

7.4 Data recorded on Form 101-01 (Attachment B) is used to document proper balance operation by performing a weight cross-check. Place the first set of required weights, listed on the form under the “Left-hand Pan” column, on the left-hand pan. Place the first set of required weights, listed on the form under the “Right-hand Pan” column, on the right-hand pan. Each weight should be placed near the center of the pan.

The meter needle should be at center-scale position. If the needle is at center-scale position, place a check on Form 101-01 for that set of weights.

If the needle is not at center-scale for any of the weights sets, contact the C&M team leader.

If the meter is at center-scale, continue placing the required weight sets on the balance until the largest weight to be used has been cross-checked. At the conclusion of the balance cross-check, sign and date Form 101-01.

7.5 Turn on the Macintosh “Gravimetric Gas Blending” computer. Position the mouse on the "Gravimetric Gas Blending Program " icon and double-click.

The “Gravimetric Gas Blending Program” start-up screen will appear as shown in Attachment C. Click on the “Enter Data” button.

7.6 The computer will display the "Gravimetric Gas Blending Input Data" screen. Enter the following data in the corresponding spaces:

Blending Date, Component Minor, Components Diluent, Minor Diluent Conc. (ppm), % O2 in Zero Air, and Technician ID #.

7.7 Ensure that "Cylinder A" and "Cylinder B" are connected to the manifold to perform the leak test. While performing Steps 7.7.1 through 7.7.7, refer to Attachment E.

7.7.1 Close manifold valves V1 through V9 by rotating them clockwise.

7.7.2 Locate the cylinder to be filled ("Cylinder A") and connect it to the junction of valves V3 and V7.

7.7.3 Verify that the cylinder that contains the desired diluent is connected to gas valve V4a, V4b, V4c, or V4d.

7.7.4 To obtain a desired cylinder concentration, determine the appropriate nominal parent gas concentration by referring to the "Gravimetric Gas Blending Scheme," see Attachment F. On the chart, locate the desired gas concentration. In the circle directly above the desired concentration is the nominal parent gas concentration.

The same cylinders should always be used for the same components and approximate concentrations. The only time this rule does not apply is when a new cylinder is being used.

Locate the parent gas cylinder that contains the concentration indicated on the chart and attach it to valve V6 (see Attachment E) on the gas blending manifold. This is "Cylinder B."

Note: The blending scheme shown in Attachment F is constructed to utilize at least 10 grams of the parent gas in each dilution. This is very important in order to achieve the required blend accuracy.

7.7.5 Open gas valves V2, V3, V4, and V6. Set DPC-2 to 1800 psig by rotating the thumbwheel switches to 1800.0. Slowly open valve V1, to pressurize the manifold until the 2000 psig gauge, G2, reads 1800 psig. DPC-2 will cycle on and off until the set-point pressure is obtained. Allow the system to stabilize. Stabilization is defined as no on/off action by DPC-2.

7.7.6 Check the G1 0-2000 psi gauge pressure and compare it to the indicated pressure of DPC-2. If the pressure differs by more than 20 psi, notify C&M team leader.

Close valve V1 and allow the manifold to remain in this condition for 10 minutes. Observe the reading of G1 for indications of leakage.

If the pressure change is less than 5%, proceed to Step 7.8.

7.7.7 If the pressure changes more than 5%, attempt to find and correct the leaks. Repeat Steps 7.7.5 and 7.7.6 until the reading remains within 5% of the set pressure for 10 minutes. If unable to correct the problem and meet this requirement, notify the C&M team leader.

7.8 Prepare the gas cylinder for blending. The procedure will leave the cylinder in a vacuum.

7.8.1 In the blending logbook, record the serial number of the "Cylinder A."

7.8.2 If not already on, place the fans and blower switch, S1, in the "On" position. See Attachment E for switch location.

7.8.3 Close gas valves V2 and V4, and ensure that gas valve V1 is closed.

7.8.4 Open gas valves V3, V7, and V9 and slowly vent the contents of "Cylinder A" into the hood. When the pressure is lowered to approximately 17 psia, turn on "S2" to apply power to the vacuum pump. Close valve V7, then slowly open gas valve V5. This applies a vacuum to "Cylinder A."

7.8.5 Observe the G2 meter pressure and evacuate the cylinder to less than 0.8 psia. When the pressure at G2 is reduced to less than 0.8 psia, close valve V5 and turn off the vacuum pump.

7.8.6 Set DPC-2 to 200 psi and open gas valves V4 and V1. Fill "Cylinder A" to 200 psi with the diluent to be used. When the pressure is 200 psi, close valve V4.

7.8.7 Repeat Steps 7.8.4 through 7.8.6 again and then continue with Step 7.8.8.

7.8.8 Open gas valves V3, V7, and V9 and slowly vent the contents of "Cylinder A" into the hood. When the pressure is lowered to approximately 17 psia, turn on "S2" to apply power to the vacuum pump. Close valve V7 and slowly open gas valve V5. This applies a vacuum to "Cylinder A."

7.8.9 Observe the G2 meter pressure and evacuate the cylinder to less than 0.8 psia. When the pressure at G2 is reduced to less than 0.8 psia, close valve V5, and turn off the vacuum pump. Verify that the pressure in the cylinder is equal to or less than 0.8 psia and that it remains in a vacuum.

7.8.10 Firmly close cylinder valve V9 then close gas valve V5. Verify that all valves are rotated fully clockwise (turned off). Disconnect the "Cylinder A" from the blending manifold.

8. Test Procedure

The preparation of gravimetric gas mixtures basically involves two processes - filling and weighing. To achieve target concentrations, target pressures are derived according to the filling data calculated from what is known as the "Partial Pressure Formula". However, the actual concentrations are based on the weights of components determined gravimetrically.

Refer to Attachment G for calculation procedures. Test data are entered into the "Gravimetric Gas Blending Program."

Since the relative weighing error is greater for small amounts, a stepwise dilution system is used for blending to minimize error. Pure gas components are diluted to make a working "Parent" or "Minor" blend.

100 Weighing "Cylinder A

Note: If during the weighing process the meter needle reads to the right of center-scale (zero), weight must be added to the left-hand pan. That weight must be used during the weighing process.

101 Wipe "Cylinder A" (which has been evacuated) with a clean shop towel to remove any dust from it and place it on the left-hand pan of the balance.

102 Place a similar type cylinder (tare cylinder) on the right-hand pan. The same tare cylinder must be used for all subsequent "Cylinder A" weighing.

103 Carefully unlock the pans and beam. Allow the balance reading to stabilize. Observe the micro-amp meter on the right-hand side of the balance. If the needle is pointing to the left of center-scale (zero), weight must be added to the right-hand pan. Add and/or remove combinations of weights to the right-hand pan until adding a 1-gram weight causes the needle to move from left of center-scale to right of center-scale. This will indicate that the weight is within 1 gram of the desired center-scale reading.

104 Remove the 1-gram weight from the right-hand pan and close the balance door.

Rotate the vernier mechanism knob to the right until the meter needle reaches the center-scale position. Allow the balance to stabilize.

105 Lock and unlock the pans to ensure the repeatability of the scale reading. Verify that the meter needle remained at center-scale.

106 Lock the pans and remove "Cylinder A" from the left-hand pan.

107 At the computer, on the first line under the "New Blend" heading, type the "Cylinder A" serial number. Continue across on the first line and type "Vent" under the "Parent Blend" heading. This removes the "Old Cylinder" data from the "Gravimetric Cylinder Data Base."

108 On the second line type the following:

under the "New Blend" heading, the "Cylinder A" serial number

under the "Parent Blend" heading, "Pure" or the "Bottle Number"

under the "Initial Empty" heading, type the total weight that was added to the right-hand pan plus the vernier scale reading.

109 Locate the "Cylinder A" number in the blending logbook and record the total weight that was added to the right-hand pan plus the vernier scale reading.

200 Adding and Weighing Parent Gas

Note: If during the weighing process the meter needle reads to the right of center-scale (zero), weight must be added to the left-hand pan. That weight must be used during the weighing process.

201 Attach "Cylinder A" to the blending manifold.

202 Rotate the thumbwheel switches of DPC-1 to set the pressure to 200 psi.

203 Open gas valves V2, V3, and V8.

204 Purge the line from parent minor gas "Cylinder B" to "Cylinder A" by slowly opening gas valve V6 and allowing the parent gas to flow.

205 Close valve V6 and open gas valve V7 until the pressure is lowered to approximately 20 psi on the G1 meter. Immediately close gas valve, V7.

206 Repeat Steps 203 through 205 two more times then continue with Step 207.

207 Determine the required pressure of the parent gas by performing the following calculation:

$$P_{PAR} = \frac{Conc_{Des} * P_{Final}}{Conc_{PAR}}$$

Where: P_{PAR} = calculated pressure of parent gas to be added
$Conc_{Des}$ = desired final concentration in parts per million (ppm)
P_{Final} = final desired new blend pressure after diluent is added (usually 1500 psia)
$Conc_{PAR}$ = parent concentration in ppm from the actual cylinder

All pressures are in pounds per square inch, absolute (psia)

208 Rotate the DPC-1 thumbwheel switches to set the pressure to the pressure calculated in Step 207.

209 Slowly open valve V6 and allow pressure to build in the line.

210 Slowly open valve V9, allow the "Cylinder A" to fill.

211 When filled, close valve V9 and then valve V8. Allow the system to stabilize.

212 Open valve V7 to allow the pressure at V7 to go to zero, then close valves V2 and V7.

213 Remove "Cylinder A" from the blending manifold and place it on the left-hand pan of the balance and rotate the vernier chain to zero.

214 Carefully unlock the pans and beam. Allow the balance reading to stabilize. Observe the micro-amp meter on the right-hand side of the balance. If the needle is pointing to the left of center-scale (zero), weight must be added to the right-hand pan. Add and/or remove combinations of weights to the right-hand pan until adding a 1-gram weight causes the needle to move from left of center-scale to right of center-scale. This will indicate that the weight is within 1 gram of the desired center-scale reading.

215 Remove the 1-gram weight from the right-hand pan and close the balance door.

Rotate the vernier mechanism knob to the right until the meter needle reaches the center-scale position. Allow the balance to stabilize.

216 Lock and unlock the pans to ensure the repeatability of the scale reading. Verify that the meter needle remained at center-scale.

217 At the computer, under the "After Adding Minor" heading on the corresponding line, type in the total weight that was added to the right-hand pan plus the vernier scale reading.

Note: The weight of the parent gas added should be nominally greater than 10 grams. Any weight greater than 10 grams will have an accuracy better than 1 part in 10000 or 0.01%.

218 Locate the bottle number in the blending logbook and record the total weight that was added to the right-hand pan plus the vernier scale reading.

300 Adding, Blending, and Weighing Diluent Gas

Note: If during the weighing process the meter needle reads to the right of center-scale (zero), weight must be added to the left-hand pan. That weight must be used during the weighing process.

301 "Cylinder A" is now ready for the addition of the diluent gas. Attach "Cylinder A" to the blending manifold at the junction of valves V3 and V7.

302 Rotate the DPC-2 thumbwheel switches to set the final pressure, normally 1500 psia.

303 Open the V4-1-d valve for the diluent being used. Slightly open valve, V1 and allow the pressure to build to the setting of DPC-2.

304 Close valve V1 and then open valve V7. This will purge the line between the diluent gas cylinder and "Cylinder A." Observe G1 and allow the line to purge until the pressure is reduced to 20 psi.

305 Close valve V7 while the gas is flowing. Repeat Steps 303 through 305 two more times and then continue with Step 306.

306 Open valve V1 and, when gauge G1 reaches a pressure higher than that in "Cylinder A." Slowly open "Cylinder A" valve V9 and fill the cylinder with diluent.

307 When the target pressure is obtained, DPC-2 will stop gas flowing. Close gas valves, V1, V3 and V9. Do not remove the cylinder.

308 If the cylinder is aluminum, allow it to cool for approximately 30 minutes. If it is steel, the cooling time should be approximately 1 hour.

Note: Ensure that the pressure of the diluent cylinder is always higher than the pressure of the cylinder being filled so as not to cause gas to back flow from the cylinder being filled to the diluent cylinder.

309 When cylinder cooling is complete, fill it again by repeating steps 306 and 307.

310 After the pressure is stabilized, close valves V9, V4, and V1. Open valve V7 to release the pressure and contents of the manifold line, then close valves V3, V7, and V4.

311 Remove "Cylinder A" from the blending manifold and place it on the left-hand pan of the balance and rotate the vernier chain to zero.

312 Carefully unlock the pans and beam. Allow the balance reading to stabilize. Observe the micro-amp meter on the right-hand side of the balance. If the needle is pointing to the left of center-scale (zero), weight must be added to the right-hand pan. Add and/or remove combinations of weights to the right-hand pan until adding a 1-gram weight causes the needle to move from left of center-scale to right of center-scale. This will indicate that the weight is within 1 gram of the desired center-scale reading.

313 Remove the 1-gram weight from the right-hand pan and close the balance door. Rotate the vernier mechanism knob to the right until the meter needle reaches the center-scale position. Allow the balance to stabilize.

314 Lock and unlock the pans to ensure the repeatability of the scale reading. Verify that the meter needle remained at center-scale. If it did not, repeat Steps 312 through 314 again. If you still can not obtain a center-scale reading, check for leaks.

315 At the computer, on the second line under the "After Adding Diluent" heading, type the total weight that was added to the right-hand pan plus the vernier scale reading.

316 Locate the bottle number in the blending logbook and record the total weight that was added to the right-hand pan (or left-hand if the meter was reading to the right of center-scale) plus the vernier scale reading.

317 Press the "Process Gas Blend Data" button.

9. Data Input

The "Blending Date," "Components," "Operator's ID Number," "Cylinder Numbers," "Cylinder Weights", and "Comments" are entered in the "Gravimetric Gas Blends Input Data" screen while performing the procedure.

10. Data Analysis

10.1 Verify that "Gravimetric Gas Blends Analysis" report has the following information:

Cylinder serial number

Parent blend

Blending weights

Blend components

10.2 Print the "Gravimetric Gas Blend Analysis" by pressing the "Gravimetric Cylinder Blender Program Print Gas Blend Report" button and review it for raw data used, the mass ratio of the components, and the concentration of the blend. This printout also has an updated inventory showing the current stock of gravimetric blends by composition and cylinder serial number.

Verify that the initial weights listed are the same as those that appear in the log book.

10.3 If a gas cylinder was previously used, verify that the previous data were deleted from the "Gravimetric Cylinder Data Base."

10.4 Verify that the data was entered properly by comparing the "Gravimetric Cylinder Data Base" values to the entries in the log book.

If the report data entries are correct, click on the "Update Gravimetric Cylinder Data Base" button of the "Gravimetric Gas Blending Program." This automatically stores the data in the computer and updates the "Gravimetric Cylinder Data Base."

Click on the "Print Gravimetric Cylinder Data Base" button of the "Gravimetric Cylinder Blender Program" to print an updated version of the "Gravimetric Cylinder Data Base."

10.5 Ensure that the new cylinder concentration is within ±5 % of the concentration of the cylinder being replaced or of the desired new gas requirement.

If the newly filled cylinder meets the above requirements, complete the "Gravimetric Cylinder Identification Tag" (see Attachment I) and affix it to the cylinder.

If the newly filled cylinder does not meet the 5% requirement, repeat this procedure.

Place the copy of the new version of the "Gravimetric Gas Blend Analysis" report in the Gas Lab file.

11. Data Output

11.1 The following information is stored in the data base when the "Update Gravimetric Cylinder Data Base" button is selected:

Cylinder serial number

Blend components

Molecular weights of the components

Blend concentrations

Mass ratios

Cylinder pressures

Blending date

11.2 A printed copy of the updated Gravimetric Cylinder Data Base is placed in the C&M file.

12. Acceptance Criteria

12.1 The total weight of the parent blend added to the cylinder must be more than 10 grams.

12.2 The same cylinders should always be used for the same components and approximate concentrations. The only time this rule does not apply is when a new cylinder is being used.

12.3 The pressure change of the leak test of Step 7.9 must be less than 5 %.

13. Quality Control Provisions

13.1 The balance and weight cross-check shall be performed and accepted as described in Section 7 at least every three months.

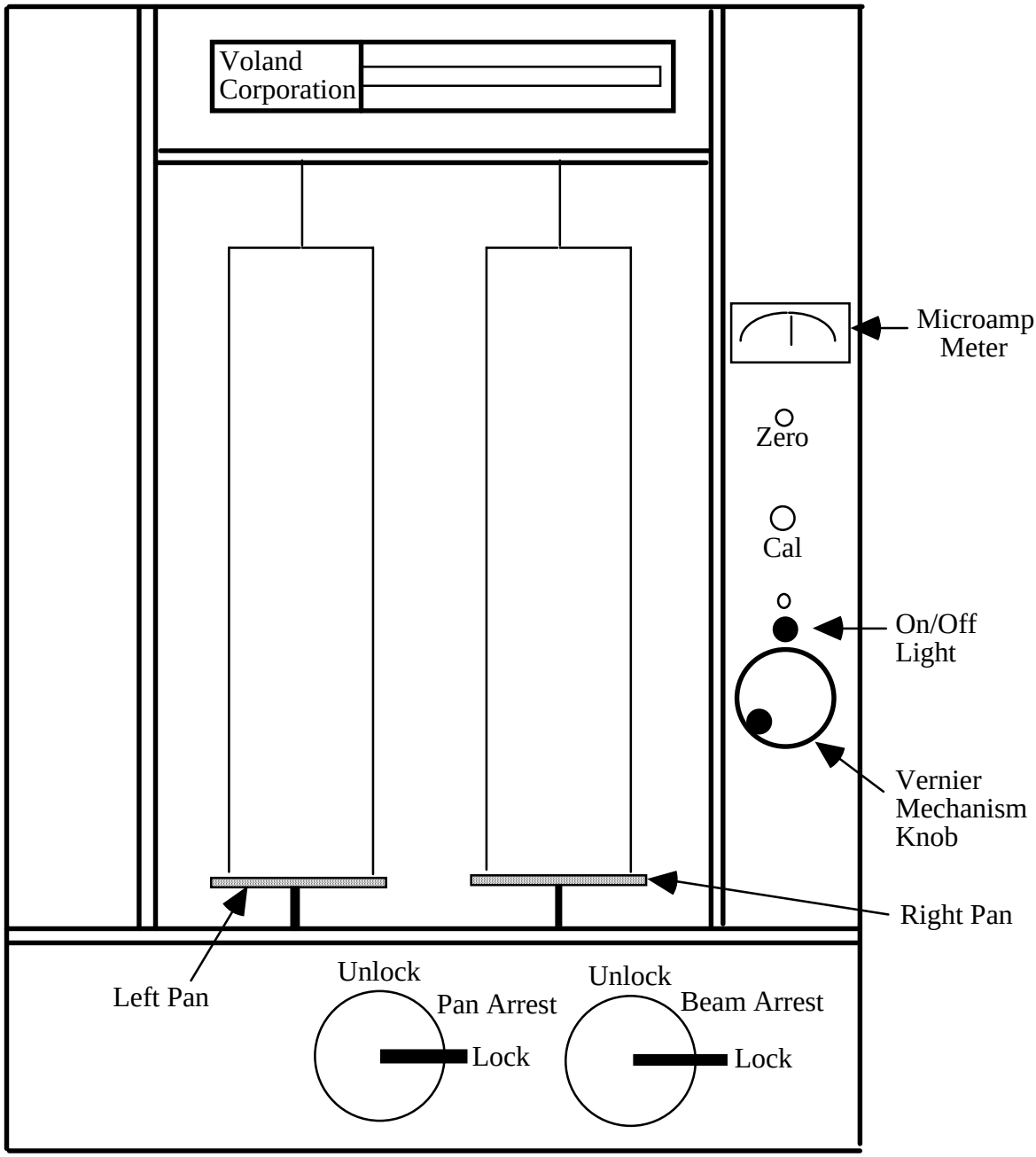
13.2 NIST-certified weights are used to check the working weights at 6-month intervals.

13.3 A cylinder leak check is performed.

13.4 The newly-blended gravimetric gas standards are correlated with other gravimetric gas standards and NIST gas standard reference materials before the just blended gravimetric cylinder is used for naming secondary gas standards.

Attachment A

Voland Scale



Attachment B

Weight Cross-check Worksheet

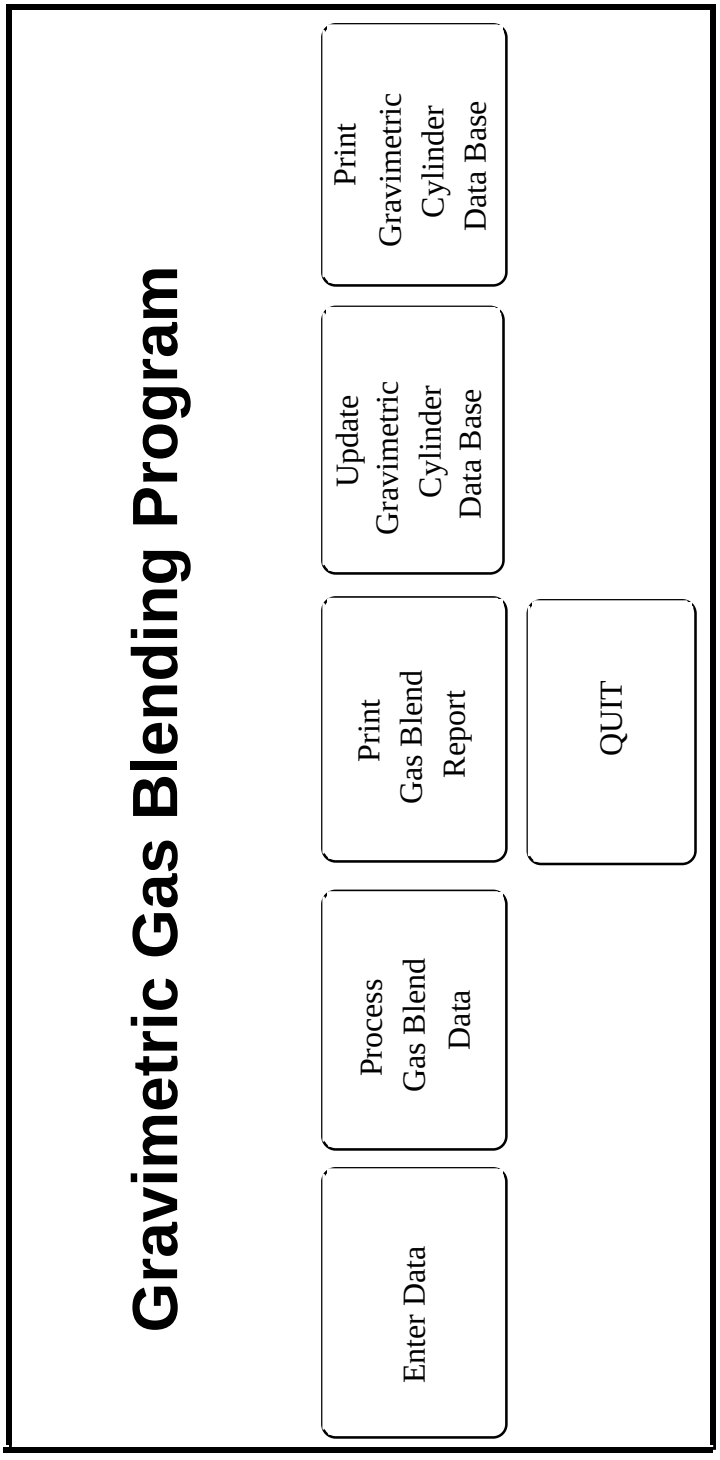
Place the required combination of weights listed under the "Left-hand Pan" column on the left pan. Place the weight listed under the "Right-hand Pan" on the right-hand pan.

Left-hand Pan Weights (grams)	Right-hand Pan Weights (grams)	Within ±0.003 grams
0.0	0.0	_____
1.0	Vernier	_____
1.0 + 2.0	3.0	_____
2.0 + 3.0	5.0	_____
2.0 + 3.0 + 5.0	10.0	_____
2.0 + 3.0 + 5.0 + 10.0	20.0	_____
10.0 + 20.0	30.0	_____
20.0 + 30.0	50.0	_____
20.0 + 30.0 + 50.0	100.0	_____
20.0 + 30.0 + 50.0 + 100.0	200.0	_____
100.0 + 200.0	300.0	_____
200.0 + 300.0	500.0	_____
200.0 + 300.0 + 500.0	1000.0	_____

I have performed the steps in accordance with the requirements of TP 101.

Technician's Name _____ Date _____

Attachment C



Attachment D

Gravimetric Gas Blending
Input Data

Blending Date	Component Minor	Component Diluent	Minor Diluent Conc. (ppm)	% O2 in Zero Air	Technician ID #	Process Date/Time

Blend No.	Cylinder Numbers New Blend	Parent Blend	Cylinder Weights (Grams) Initial Empty	After Adding Minor	After Adding Diluent	Comments
1						
2						
3						
4						
5						
6						

Component Symbols

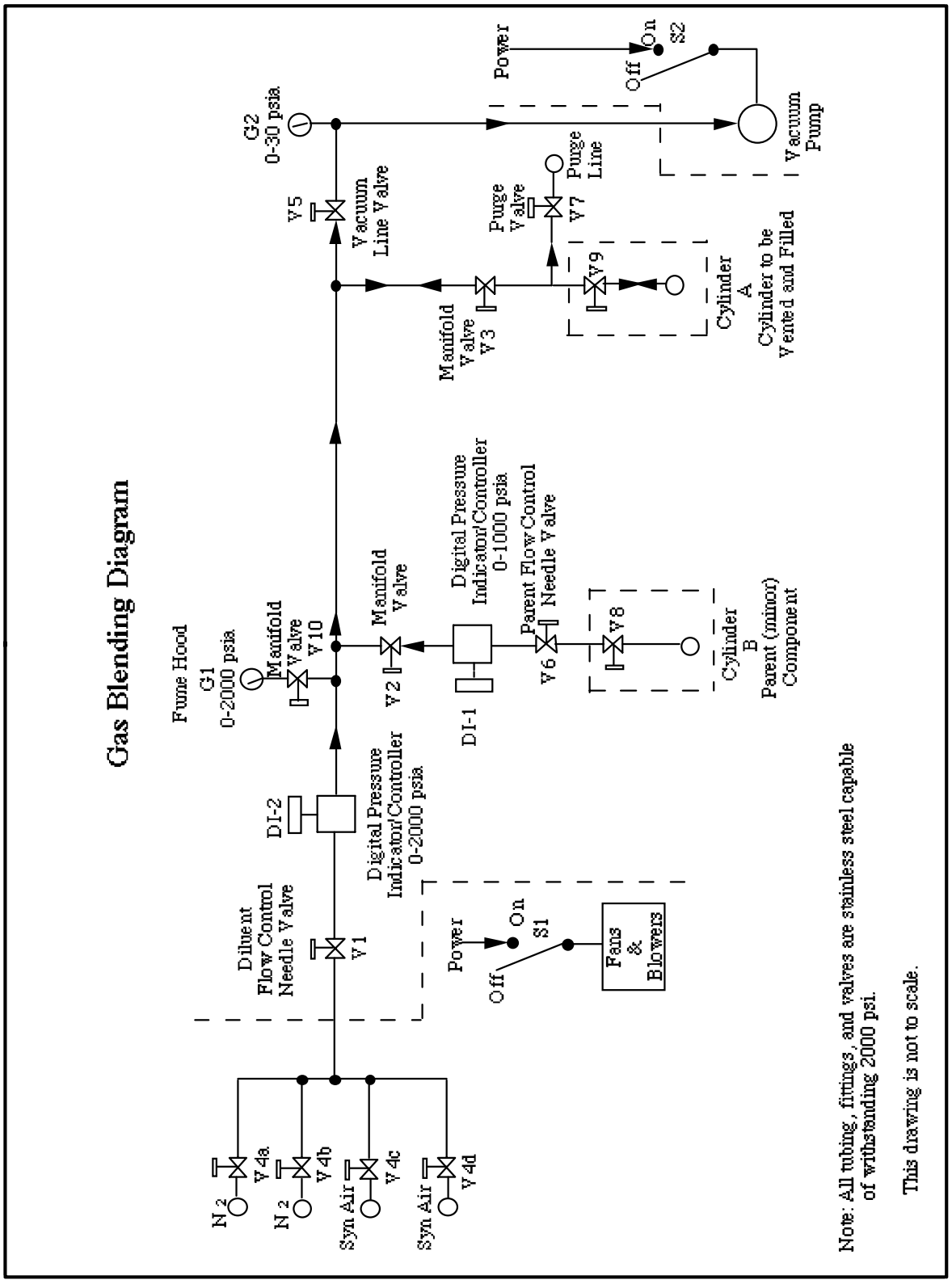
Gasses:
- C3H8 - Propane
- CO - Carbon Monoxide
- CO2 - Carbon Dioxide
- NO - Nitric Oxide
- CH4 - Methane

Minors:
- SO2 - Sulfur Dioxide
- O2 - Oxygen
- HE - Helium
- H2 - Hydrogen

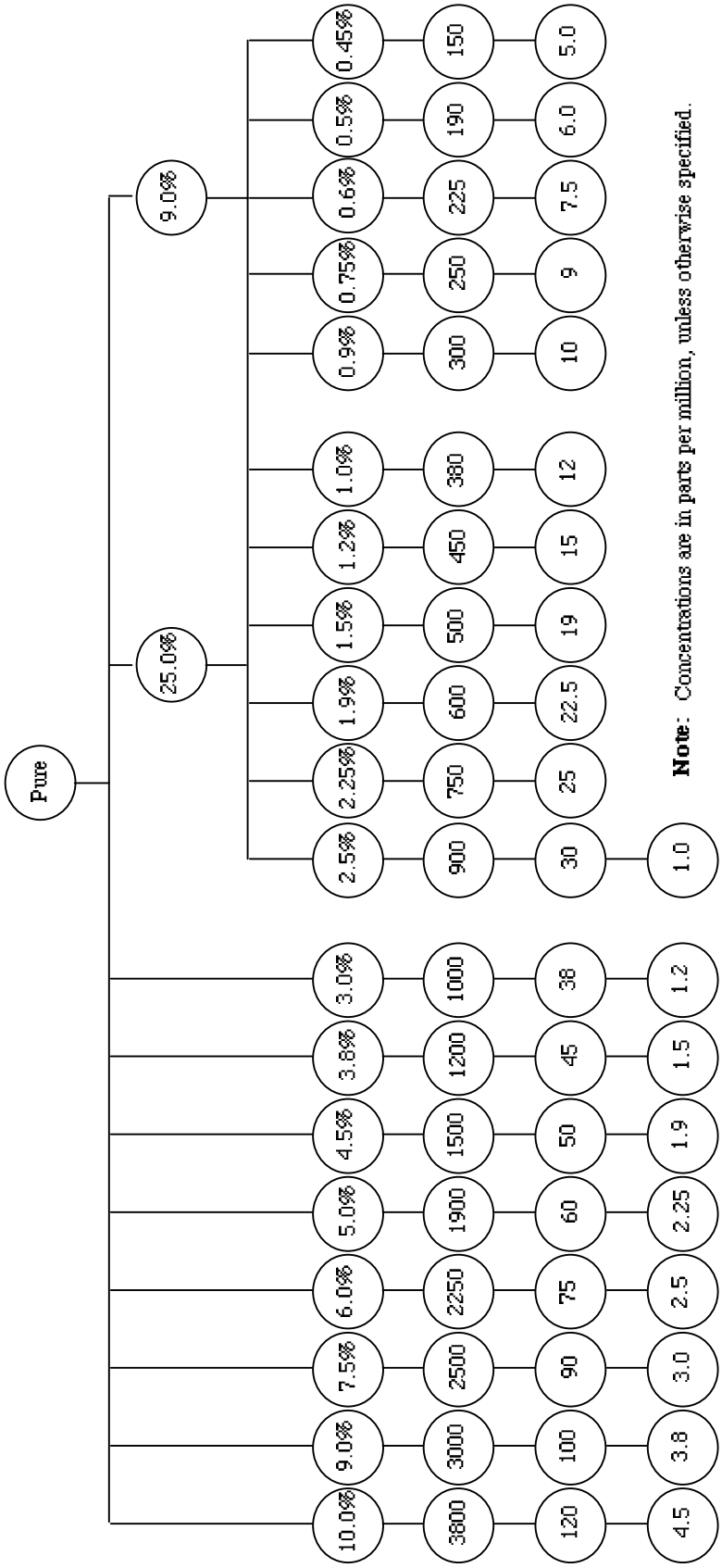
Diluents:
- Air - Zero Grade (synth. blend)
- N2 - Ultra Pure Nitrogen

Notes:
Two special parent cylinder numbers are recognized.
"Pure" indicates a parent blend has been made from pure components.
"Vent" indicates that the cylinder reference has been vented and is to be deleted from the file of cylinders.

Attachment E



Attachment F



Attachment G

Gravimetric Gas Blending Calculations

Gas Data Base

$MW_{N2} := 28.0134$	$N2_{conc} := 1000000$	Ultra Pure Nitrogen
$MW_{CH4} := 16.0426$	$CH4_{conc} := 1000000$	Methane
$MW_{C3H8} := 44.0962$	$C3H8_{conc} := 1000000$	Propane
$MW_{CO} := 28.0104$	$CO_{conc} := 1000000$	Carbon Monoxide
$MW_{NO} := 30.0061$	$NO_{conc} := 1000000$	Nitric Oxide
$MW_{SO2} := 64.0588$	$SO2_{conc} := 1000000$	Sulphur Dioxide
$MW_{CO2} := 44.0098$	$CO2_{conc} := 100$	Carbon Dioxide
$MW_{O2} := 31.9988$	$O2_{conc} := 100$	Oxygen
$MW_{HE} := 4.0026$	$HE_{conc} := 100$	Helium
$MW_{H2} := 2.0158$	$H2_{conc} := 100$	Hydrogen
$MW_{AIR} := 28.8503$	$AIR_{conc} := 1000000$	Zero Grade (Synthetic Blend) Air

Inputs

Parent Cylinder is HH-10600 - Its Minor Component is C3H8 (Propane) - Its diluent is N2
Diluent gas is Air - Component is N2 (Nitrogen)

$Conc_{minor.in.diluent} := 0$	Minor Concentraton in the Diluent Cylinder
$Percent_{O.2} := 21$	Percent Oxygen in Zero Air
$Cylinder_{Initial} := 69.183$	Empty Cylinder Weight, grams
$Cylinder_{parent.gas} := 85.065$	Cylinder Weight after adding Minor (Parent Gas)
$Cylinder_{parent.and.diluent.gas} := 513.108$	Cylinder Weight after adding Minor and Diluent
$PMR := 0.20749$	Parent Cylinder (HH-10600) mass ratio (from data base
$MW_{diluent} := MW_{AIR}$	Mole Weight of the diluent gas in the diluent cylinder
$MW_{minor} := MW_{C3H8}$	Mole Weight of the minor gas in the parent cylinder
$MW_{diluent.in.parent} := MW_{N2}$	Mole Weight of the diluent in the parent cylinder
$minor_{conc} := C3H8_{conc}$	Concentration units of the minor gas

(This document follows the presentation in "Procedure for Makinng Gravimetric Binary Gas Mixtures", by C. D. Paulsell, EPA, 1 August 1973)

Attachment G continued

Gravimetric Gas Blending Calculations

First determine the weight of the parent and diluent gases that were put into the blended gas cylinder.

$mass_{parent.gas} := Cylinder_{parent.gas} - Cylinder_{Initial}$ $mass_{parent.gas} = 15.882$

$mass_{diluent.gas} := Cylinder_{parent.and.diluent.gas} - Cylinder_{parent.gas}$ $mass_{diluent.gas} = 428.043$

If the diluent contains a concentration of the minor, this must be corrected in the final concentration. A diluent mass ratio (DMR) must be computed for the diluent cylinder.

$$DMR := \frac{-MW_{minor} \cdot Conc_{minor.in.diluent}}{MW_{diluent} \cdot Conc_{minor.in.diluent} - MW_{minor} \cdot Conc_{minor.in.diluent} - MW_{diluent} \cdot 10^6}$$

$DMR = 0$

If the parent cylinder is not a pure gas, then the mass ratio of the parent must be known. From the PMR (parent mass ratio) the mass of the minor can be determined as follows:

$PMR = 0.20749$

$mass_{parent.gas} = 15.882$

$mass_{diluent.gas} = 428.043$

$mass_{total.gas} := mass_{parent.gas} + mass_{diluent.gas}$ $mass_{total.gas} = 443.925$

$mass_{minor.in.blend} := mass_{parent.gas} \cdot PMR + mass_{diluent.gas} \cdot DMR$ $mass_{minor.in.blend} = 3.295$

$mass_{diluent.in.blend} := mass_{parent.gas} \cdot (1 - PMR) + mass_{diluent.gas} \cdot (1 - DMR)$

$mass_{diluent.in.blend} = 440.63$

$mass_{total.blend} := mass_{minor.in.blend} + mass_{diluent.in.blend}$ $mass_{total.blend} = 443.925$

(This document follows the presentation in "Procedure for Makinng Gravimetric Binary Gas Mixtures", by C. D. Paulsell, EPA, 1 August 1973)

Attachment G continued

Gravimetric Gas Blending Calculations

The following is a clarification of the Mole Weights to be used in computations.

For the diluent "air," an adjustment to the molecular weight must be made on the basis of O_2 concentration in the zero air. The molecular weight of air is given as:

$$MW_{AIR} := \frac{Percent_{O.2} \cdot MW_{O2} + (100 - Percent_{O.2}) \cdot MW_{N2}}{100} \qquad MW_{AIR} = 28.85033$$

$$MW_{diluent} := MW_{AIR} \qquad MW_{diluent} = 28.85$$

$$MW_{minor} = 44.096 \qquad MW_{C3H8} = 44.096$$

$$MW_{diluent.in.parent} = 28.013 \qquad MW_{N2} = 28.013$$

The masses and mole weights are used to determin the molar masses as follows:

$$moles_{minor.in.blend} := \frac{mass_{minor.in.blend}}{MW_{minor}} \qquad moles_{minor.in.blend} = 0.07473$$

$$moles_{diluent.in.blend} := \frac{mass_{parent.gas} \cdot (1 - PMR)}{MW_{diluent.in.parent}} + \frac{mass_{diluent.gas} \cdot (1 - DMR)}{MW_{diluent}}$$

$$moles_{total.blend} := moles_{minor.in.blend} + moles_{diluent.in.blend} \qquad moles_{total.blend} = 15.361$$

Now determine the mass ratio and concentration of the blended gas.

$$MR := \frac{mass_{minor.in.blend}}{mass_{total.blend}} \qquad MR = 0.007423$$

$$minor_{conc} = 1 \cdot 10^{6}$$

$$Conc_{blend} := \frac{moles_{minor.in.blend}}{moles_{total.blend}} \cdot minor_{conc} \qquad Conc_{blend} = 4865.078$$

Verify computations be recomputing Mole Weights and calculate the Mole Weight of the blended gas.

$$MW_{minor.in.blend} := \frac{mass_{minor.in.blend}}{moles_{minor.in.blend}} \qquad MW_{minor.in.blend} = 44.096$$

$$MW_{diluent.in.blend} := \frac{mass_{diluent.in.blend}}{moles_{diluent.in.blend}} \qquad MW_{diluent.in.blend} = 28.826$$

$$MW_{total.blend} := \frac{mass_{total.blend}}{moles_{total.blend}} \qquad MW_{total.blend} = 28.9$$

(This document follows the presentation in "Procedure for Makinng Gravimetric Binary Gas Mixtures", by C. D. Paulsell, EPA, 1 August 1973)

Attachment G continued

Gravimetric Gas Blending Calculations

Computation of the mass and concentration of Oxygen in the new blended cylinder.

$$mass_{O2.parent.gas} := \frac{mass_{parent.gas} \cdot (1 - PMR) \cdot \left(\frac{1}{MW_{diluent.in.parent}} - \frac{1}{MW_{N2}}\right)}{\left(\frac{1}{MW_{O2}} - \frac{1}{MW_{N2}}\right)} \qquad mass_{O2.parent.gas} = 0$$

$$mass_{O2.diluent.gas} := \frac{mass_{diluent.gas} \cdot (1 - DMR) \cdot \left(\frac{1}{MW_{diluent}} - \frac{1}{MW_{N2}}\right)}{\left(\frac{1}{MW_{O2}} - \frac{1}{MW_{N2}}\right)} \qquad mass_{O2.diluent.gas} = 99.699$$

$$mass_{O2.in.blend} := mass_{O2.parent.gas} + mass_{O2.diluent.gas} \qquad mass_{O2.in.blend} = 99.699$$

$$Conc_{O2.in.blend} := \frac{100 \cdot \left(\frac{mass_{O2.in.blend}}{MW_{O2}}\right)}{moles_{total.blend}} \qquad Conc_{O2.in.blend} = 20.284$$

(This document follows the presentation in "Procedure for Making Gravimetric Binary Gas Mixtures", by C. D. Paulsell, EPA, 1 August 1973)

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Since the actual blending of cylinders requires pressures to be measured to obtain the approximate concentration desired, the pressures must be calculated. Applying the concept of partial pressure, one can find the initial pressure of the parent gas as:

$$P_{PAR} = \frac{Conc_{Des} * P_{Final}}{Conc_{PAR}}$$

Where: P_{PAR} = the calculated pressure of parent gas to be added
$Conc_{Des}$ = desired final concentration in parts per million (ppm)
P_{Final} = final desired new blend pressure after diluent is added (usually 1500 psia)
$Conc_{PAR}$= parent concentration in ppm from the actual cylinder

All pressures are in pounds per square inch, absolute (psia)

Attachment H

GRAVIMETRIC GAS BLEND ANALYSIS

DATE:
MINOR COMPONENT:
DILUENT:
MINOR CONCENTRATION IN DILUENT (PPM):
%O2 IN ZERO AIR:

MEASURED DATA

CYLINDER NO.	PARENT CYLINDER NO.	MEASURED CYLINDER WEIGHTS: EMPTY (GRAMS)	AFTER MINOR (GRAMS)	AFTER MAJOR (GRAMS)	COMMENT

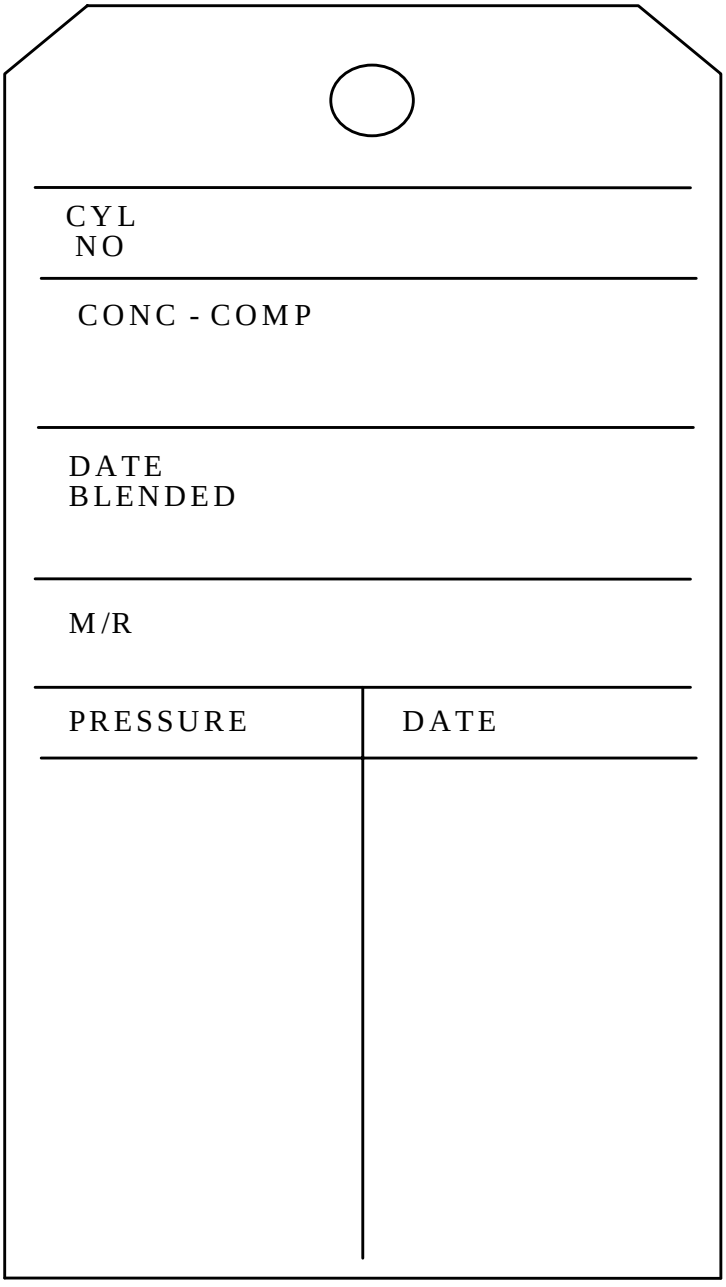
CALCULATED DATA

CYLINDER NO.	MINOR COMPONENT (GRAMS CO)	MAJOR COMPONENT (GRAMS N2)	MASS RATIO	BLEND CONCENTRATION (PPM)	OXYGEN CONCENTRATION (PCT)	QUALITY CONTROL FLAGS

PROCESSED: 08:53:01 Feb 02, 1996

Attachment I

Gravimetric Cylinder Identification Tag



Attachment J

Gravimetric Gas Cylinder Inventory						
Cylinder Number	**Minor Gas**	**Diluent Gas**	**Concentration ppm**	**Parent Cylinder**	**Comment**	**Blend Date**
FF-5824	C3H8	N2	158281.375	PURE	-	1/8/92 0:00
HH-10600	C3H8	N2	148448.5625	PURE	-	9/7/94 0:00
D-14012	C3H8	N2	65682.4375	PURE	-	9/15/94 0:00
D-14092	C3H8	N2	29895.332	PURE	-	4/7/87 0:00
HH-10681	C3H8	N2	20271.9336	PURE	-	3/20/87 0:00
F-1368	C3H8	N2	16113.6289	FF-5824	-	2/8/90 0:00
F-1361	C3H8	N2	13407.8047	HH-10600	-	9/14/94 0:00
G-11871	C3H8	N2	9532.0469	HH-10600	-	1/23/92 0:00
H-89459	C3H8	N2	7501.3594	D-14012	-	2/9/90 0:00
F-1362	C3H8	N2	6446.3242	FF-5824	-	1/16/92 0:00
F-1365	C3H8	N2	4735.4023	D-14012	-	1/24/92 0:00
H-89478	C3H8	N2	3711.4602	HH-10600	-	2/20/85 0:00
D-7469	C3H8	N2	3493.7825	HH-10600	-	9/9/94 0:00
F-1374	C3H8	N2	3351.9404	HH-10643	-	3/6/87 0:00
HH-10688	C3H8	N2	3070.5706	HH-10600	-	2/21/85 0:00
HH-10625	C3H8	N2	2464.7854	HH-10643	-	6/18/85 0:00
G-11864	C3H8	N2	2143.9966	D-14092	-	4/28/87 0:00
G-11842	C3H8	N2	1823.6045	D-14092	-	3/3/87 0:00
D-7445	C3H8	N2	1404.5723	HH-10681	-	4/23/87 0:00
G-11833	C3H8	N2	994.8967	HH-10681	-	1/27/92 0:00
G-11855	C3H8	N2	863.2502	F-1368	-	2/25/87 0:00
G-11861	C3H8	N2	741.4438	D-14092	-	3/4/87 0:00
D-7395	C3H8	N2	623.1177	F-1368	-	3/11/87 0:00
G-11846	C3H8	N2	472.6951	F-1361	-	5/21/87 0:00
F-1371	C3H8	N2	371.5828	F-1362	-	3/12/87 0:00
G-11844	C3H8	N2	261.3164	G-11871	-	3/17/87 0:00
D-7405	C3H8	N2	178.9616	F-1362	-	1/22/92 0:00
D-14081	C3H8	N2	118.5381	D-7469	-	3/26/87 0:00
D-7408	C3H8	N2	96.0903	HH-10688	-	5/4/87 0:00
D-14001	C3H8	N2	84.6591	HH-10625	-	3/27/87 0:00
D-7424	C3H8	N2	74.7775	G-11864	-	1/28/91 0:00
D-7431	C3H8	N2	62.1898	D-7475	-	3/6/80 0:00
D-7448	C3H8	N2	48.2012	D-7445	-	1/29/92 0:00
D-7451	C3H8	N2	37.0474	G-11861	-	3/6/80 0:00
D-7453	C3H8	N2	23.9534	D-7395	-	1/29/92 0:00
D-7463	C3H8	N2	19.7816	D-7472	-	3/6/80 0:00
D-7468	C3H8	N2	16.5824	F-1371	-	1/30/92 0:00
HH-799	CO	N2	251887	PURE	-	2/23/93 0:00
HH-618	CO	N2	98273.8125	PURE	-	2/12/93 0:00
D-14025	CO	N2	89300.375	PURE	-	1/2/91 0:00
D-14036	CO	N2	80060.375	PURE	-	2/18/93 0:00
HH-2162	CO	N2	69684.1875	PURE	-	2/17/93 0:00
D-1156	CO	N2	57772.3672	PURE	-	12/28/90 0:00
D-14013	CO	N2	48583.0195	PURE	-	1/3/91 0:00
D-14037	CO	N2	42481.0273	PURE	-	1/4/91 0:00
D-14060	CO	N2	37001.3906	HH-799	-	1/4/91 0:00
D-14061	CO	N2	30902.3867	HH-799	-	1/7/91 0:00
D-14062	CO	N2	23908.8516	HH-799	-	2/9/93 0:00